

Unused hydrocarbon-based insulating liquids — Test methods for evaluating the oxidation stability

The European Standard EN 61125:1993, with the incorporation of amendment A1:2004, has the status of a British Standard

ICS 17.220.99; 29.035.40

Cooperating organizations

The European Committee for Electrotechnical Standardization (CENELEC), under whose supervision this European Standard was prepared, comprises the national committees of the following countries:

Austria	Italy
Belgium	Luxembourg
Denmark	Netherlands
Finland	Norway
France	Portugal
Germany	Spain
Greece	Sweden
Iceland	Switzerland
Ireland	United Kingdom

This British Standard, having been prepared under the direction of the Cables and Insulation Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 15 July 1993

© BSI 28 July 2004

The following BSI references relate to the work on this British Standard:
Committee reference CIL/10
Draft for comment 80/22508 DC

ISBN 0 580 21870 8

Amendments issued since publication

Amd. No.	Date	Comments
15236	28 July 2004	Change to subclause 1.9.6

National foreword

This British Standard has been prepared under the direction of the Cables and Insulation Standards Policy Committee and is the English language version of EN 61125:1993, *Unused hydrocarbon-based insulating liquids — Test methods for evaluating the oxidation stability*, including amendment A1:2004, published by the European Committee for Electrotechnical Standardization (CENELEC). It is identical with IEC 1125:1992, including amendment 1, published by the International Electrotechnical Commission (IEC).

This standard describes three test methods A, B and C for evaluating the oxidation stability of mineral insulating oils and of hydrocarbon-based insulating liquids.

Method A describes a method for evaluating the oxidation stability of unused uninhibited mineral insulating oils under accelerated conditions. It supersedes IEC 74:1963, which is technically equivalent to BS 2000-307:1983, *Methods of test for petroleum and its products — Part 307: Oxidation stability of mineral insulating oil*. BS 2000-307:1983 is withdrawn.

Method B describes a method for evaluating the oxidation stability of unused inhibited mineral insulating oils under accelerated conditions. It supersedes IEC 474:1974, which has no corresponding British Standard.

Method C describes a method for evaluating the oxidation stability of unused uninhibited and inhibited hydrocarbon insulating liquids under accelerated conditions. It supersedes IEC 813:1985 (HD 486 S1:1987), which has no corresponding British Standard.

WARNING. This British Standard calls for the use of substances and procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 27 and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

English version

Unused hydrocarbon-based insulating liquids — Test methods for evaluating the oxidation stability

(includes amendment A1:2004)

(IEC 61125:1992)

Isolants liquides neufs à base
d'hydrocarbures — Méthodes d'essai pour
évaluer la stabilité à l'oxydation
(inclut l'amendement A1:2004)
(CEI 61125:1992 + A1:2004)

Neue Isolierflüssigkeiten auf Mineralölbasis —
Prüfverfahren zur Beurteilung der
Oxidationsbeständigkeit
(enthält Änderung A1:2004)
(IEC 61125:1992 + A1:2004)

This European Standard was approved by CENELEC on 1993-03-09; amendment A1 was approved by CENELEC on 2004-06-01. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CENELEC member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CENELEC member into its own language and notified to the Central Secretariat has the same status as the official versions.

CENELEC members are the national electrotechnical committees of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

CENELEC

European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B-1050 Brussels

Foreword

The text of document 10(CO)263, as prepared by IEC technical committee No. 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote in November 1991.

The reference document was approved by CENELEC as EN 61125 on 9 March 1993.

This European Standard supersedes HD 486 S1:1987.

The following dates were fixed:

- latest date of publication of an identical national standard (dop) 1994-03-01
- latest date of withdrawal of conflicting national standards (dow) 1994-03-01

Annexes designated “normative” are part of the body of the standard. In this standard, Annex A, Annex B and Annex ZA are normative.

Foreword to amendment A1

The text of document 10/577/FDIS, future amendment 1 to IEC 61125:1992, prepared by IEC TC 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as amendment A1 to EN 61125:1993 on 2004-06-01.

The following dates were fixed:

- latest date by which the amendment has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2005-03-01
- latest date by which the national standards conflicting with the amendment have to be withdrawn (dow) 2007-06-01

Contents

	Page
Foreword	2
Section 1. General	
1.1 Scope	3
1.2 Normative references	3
1.3 General principle of the methods	3
1.4 Equipment	3
1.5 Reagents	5

1.6 Cleaning of test vessels	6
1.7 Catalyst	6
1.8 Insulating liquid sample conditioning	6
1.9 Determinations on the oxidized insulating liquid	6
Section 2. Method A	
2.1 Outline of the method	9
2.2 Test conditions	9
2.3 Procedure	9
2.4 Determinations on the oxidized oil	9
2.5 Report	10
2.6 Precision	10
Section 3. Method B	
3.1 Outline of the method	11
3.2 Test conditions	11
3.3 Procedure	11
3.4 Determinations on the oxidized oil (optional)	12
3.5 Report	12
3.6 Precision	13
Section 4. Method C	
4.1 Outline of the method	15
4.2 Test conditions	15
4.3 Procedure	15
4.4 Determinations on the oxidized insulating liquid	16
4.5 Report	16
4.6 Precision	16
Annex A (normative) Determination of maximum diameter of the pores of a fritted glass filter	17
Annex B (normative) Thermometer specifications	18
Annex ZA (normative) Other international publications quoted in this standard with the references of the relevant European publications	25
Figure 1 — Typical 8 hole (4 × 2) aluminium heating block	19
Figure 2 — Aluminium alloy temperature measuring block	20
Figure 3 — Position of the tube in the oil bath	20
Figure 4 — Oxidation tube or absorption tube	21
Figure 4b — Oxidation tube and absorption tube assembly	22
Figure 5 — Soap bubble flowmeter	23
Figure 6 — Determination of maximum diameter of the pores of a glass filter	24

Section 1. General

1.1 Scope

This International Standard describes three test methods using the same apparatus for evaluating the oxidation stability of mineral insulating oils and of hydrocarbon-based insulating liquids. It comprises four sections as follows:

Section 1 describes the following items common to the three methods:

- the equipment and reagents;
- the cleaning of the glassware;
- the preparation of the catalyst and of the insulating liquid sample to be tested;
- the determinations on the oxidized insulating liquid.

Section 2 — Method A describes a method for evaluating the oxidation stability of unused uninhibited mineral insulating oils under accelerated conditions.

Section 3 — Method B describes a method for evaluating the oxidation stability of unused inhibited mineral insulating oils under accelerated conditions.

Section 4 — Method C describes a method for evaluating the oxidation stability of unused uninhibited and inhibited hydrocarbon insulating liquids under accelerated conditions.

1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

IEC 247:(1978), *Measurement of relative permittivity, dielectric dissipation factor and d.c. resistivity of insulating liquids*.

ISO 383:(1976), *Laboratory glassware — Interchangeable conical ground joints*.

ISO 4793:(1980), *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*.

ISO/DIS 6344-1, *Coated abrasives — Grain size analysis — Part 1: Definitions, designation and principles*.

1.3 General principle of the methods

The liquid sample to be tested, through which a stream of oxygen or air is bubbled, is maintained for a given period at a given temperature, 100 °C or 120 °C, in the presence of solid copper. The resistance to oxidation is evaluated from the amount of total sludge and total acidity formed or from the time to develop a given amount of volatile acidity (induction period).

1.4 Equipment

1.4.1 Heating arrangement

A thermostatically-controlled aluminium alloy block heater or oil bath may be used to maintain the insulating liquid in the desired number of oxidation tubes at the required temperature $100\text{ °C} \pm 0,5\text{ °C}$ or $120\text{ °C} \pm 0,5\text{ °C}$ (as examples see Figure 1 and Figure 3, pages 19 and 20). This temperature shall be read on a thermometer (see Annex B) inserted in an oxidation tube to within 5 mm from the bottom; this oxidation tube shall be filled with oil up to the immersion line of the thermometer and placed in the heating bath.

The temperature of the upper surface of the thermal insulation top shall be maintained at $50\text{ °C} \pm 5\text{ °C}$ (Method A) or $60\text{ °C} \pm 5\text{ °C}$ (Methods B and C). Measure this temperature by the use of a thermometer in a drilled aluminium block (see Figure 2, page 20). The surfaces of this block, other than that against the upper surface of the heating device, are protected by suitable thermal insulation of nominal 4 mm thickness. The thermal characteristics of this insulation shall be such as to permit the specified temperatures to be achieved. This block should be placed as near to the holes as practicable and within the area of the upper surface covering the heating device.

When using an aluminium heating block, the oxidation tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short aluminium alloy collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube.

In the case of oil baths, the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath (see Figure 3, page 20).

For both types of heating device, the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In the case of slackness a 25 mm internal diameter O-ring may be placed around the tube and pressed against the thermal insulation top or inserted into the annular space between the tube and the thermal insulated top. The heating bath should be equipped with supports to hold the absorption tubes.

When in use the heater shall be shielded from direct sunlight and air draughts.

NOTE When oil baths are used, for safety reasons, it may be advisable to place them in a fume hood.

1.4.2 Test vessels

Test tubes of borosilicate or neutral glass provided with a 24/29 ground joint (see ISO 383), of the following dimensions:

	mm
— overall length	210 ± 2
— external diameter	26 ± 0,5
— wall thickness	1,4 ± 0,2
— height of the head	28 ± 2
— air inlet tube:	
external diameter	5,0 ± 0,4
wall thickness	0,8 ± 0,1

The test tube is fitted with a Drechsel head to which is attached the inlet tube which extends to within $2,5 \text{ mm} \pm 0,5 \text{ mm}$ from the bottom and has its end ground at an angle of 60° to the horizontal axis (see Figure 4, page 21).

1.4.3 Absorption tubes (only required for methods B and C)

These are identical to the test vessels and the distance between the axes of the two tubes shall be $150 \text{ mm} \pm 50 \text{ mm}$ (see Figure 4 and Figure 4 bis, pages 21 and 22). Connections between the test and absorption tubes should be as short as possible, of glass tubing butt-jointed to the vessels by means of short flexible sleeves (silicone rubber sleeving has been found suitable for this purpose). These tubes are mounted outside the heating device.

1.4.4 Filtering crucibles

Gooch-type crucibles with fused-in fritted glass disk according to ISO 4793 — grade P 10 porosity (pore size index $> 4 \text{ } \mu\text{m}$ and $\leq 10 \text{ } \mu\text{m}$) of, for example, 35 ml capacity.

NOTE 1 Maximum diameter of the pores can be determined in accordance with the method described in Annex A.

NOTE 2 Alternatively polymeric membrane filters may be used, provided they are compatible with oil and solvents.

Suitable membranes consist of mixture of cellulose esters (cellulose nitrate + cellulose acetate) with the following characteristics:

- pore size: $8 \text{ } \mu\text{m}$
- thickness: $150 \text{ } \mu\text{m}$
- operating temperature: 120°C in sterilizer;
 75°C under continuous filtration.

The filtration is improved by impregnating the membrane with a suitable wetting agent (e.g.: octyl ethoxylate).

1.4.5 Porcelain vessels

Porcelain crucibles, capacity: 50 ml.

NOTE Alternatively, aluminium foil pans of the same capacity may be used.

1.4.6 Soap bubble flowmeter

For measuring gas flow-rate (see Figure 5, page 23).

1.4.7 Timer

For measuring gas flow-rate. Subdivisions of the graduation: 0,2 s.

1.4.8 Gas supply

Gas (oxygen or air according to the method) from a compressed gas cylinder, is dried by passing through a scrubber bottle containing concentrated sulphuric acid and then through a tower filled with alternate layers of glass wool and soda lime.

Alternatively dessicant drying tubes may be used.

The dried gas is passed into the oxidation tube via a flow control system which shall be suitable for the specified flow-rate. This may consist of a manifold, connected to the gas-purifying train, with a number of tappings, each provided with a fine-control adjustable needle valve and supplying the gas to one oxidation tube.

The rate of gas flow may be conveniently measured by means of a soap bubble flowmeter or of a calibrated capillary tube flowmeter. In that case, the difference in level of the liquid in the two limbs of the flowmeter should be sufficiently great to ensure that adequate sensitivity of measurement is obtained over the range of gas flow-rates.

Any system known to be of equal or greater efficiency can however be used.

NOTE A two-stage pressure regulator and a pressure compensator vessel may be helpful to achieve the required accurate regulation of the gas pressure.

1.4.9 Burette

Volume 10 ml with graduations of 0,01 ml.

1.4.10 Thermometer

A thermometer conforming to the requirements given in Annex B.

1.4.11 Erlenmeyer

Erlenmeyer flasks, volume 500 ml, with ground glass stopper.

1.5 Reagents

1.5.1 Normal heptane (analytical grade)

1.5.2 Alkali blue 6 B indicator

2 g of alkali blue 6 B per 100 ml of azeotropic ethanol (about 5 % water).¹⁾

Extract 2 g of alkali blue 6 B with azeotropic ethanol in a Soxhlet apparatus, filter if necessary, and dilute to 100 ml with azeotropic ethanol.

Add 3 ml of 0,1 M HCl and after 24 h carry out a check acid value to confirm that the indicator has been sensitized, i.e. a colour change as given in 1.9.2 of this standard occurs. Should sensitization not be sufficient, then add more 0,1 M HCl until sensitization has occurred. Periodic resensitization may be necessary.

1.5.3 Phenolphthalein indicator

1 g of phenolphthalein per 100 ml of azeotropic ethanol (about 5 % water).

1.5.4 Titration solvent

Mix three parts by volume of toluene (analytical grade) and two parts by volume of azeotropic ethanol.

1.5.5 Potassium hydroxide

0,1 M alcoholic solution.

¹⁾ Alternatively isopropanol containing 5 % of water may be used.

1.5.6 Oxidant gas

1.5.6.1 Oxygen

Obtained from liquid air, 99,4 % minimum purity.

1.5.6.2 Air

Obtained from cylinder.

1.5.7 Hydrochloric acid

Aqueous 0,1 M solution.

1.5.8 Chloroform (analytical grade)

1.6 Cleaning of test vessels

The test and the absorption tubes shall be chemically cleaned. Wash with acetone followed by distilled or deionized water.

Drain and then soak in concentrated sulphuric acid for a minimum of 16 h. Drain and complete removal of acid by washing, first with tap water, then with distilled or deionized water. Dry the tubes in an air oven at 105 °C for at least 3 h, and then allow to cool to room temperature in a desiccator or a dry cabinet in which they are kept ready for use.

1.7 Catalyst

The solid copper used as oxidation catalyst consists of a wire of soft electrolytic copper, of diameter between 1 mm and 2 mm (of such a length as to give a surface area of $9,7 \text{ cm}^2 \pm 0,1 \text{ cm}^2$ for method A and $28,6 \text{ cm}^2 \pm 0,3 \text{ cm}^2$ for methods B and C). It is prepared as follows:

1.7.1 Immediately before use, the requisite length of copper wire is cleaned with P220 grade silicon carbide abrasive cloth (ISO 6344-1). All traces of abrasive are removed with a lintless filter paper and then with a dry, lintless cloth.

1.7.2 Roll the wire into a spiral of approximately 2 cm external diameter and 5 cm long.

1.7.3 The spiral is thoroughly cleaned by dipping it into normal heptane, then dried in air and immediately introduced into the test vessel.

1.7.4 To avoid contamination, the prepared coil shall be handled only with tweezers. The copper wire shall not be re-used.

1.8 Insulating liquid sample conditioning

Liquid to be tested shall be filtered through a previously dried (1 h at 105 °C) fritted glass filter (ISO 4793, grade P 10 porosity) or on membrane filters of 8 µm to remove traces of sediment, fibre and excess water. The first 25 ml of filtrate should be discarded.

1.9 Determinations on the oxidized insulating liquid

1.9.1 Sludge formation

The sludge shall be precipitated by adhering strictly to the procedure described below.

The sample of 25 g of artificially aged insulating liquid is cooled in the dark for 1 h, and is then poured into an Erlenmeyer flask.

Use 300 ml normal heptane in successive fractions to rinse out the insulating liquid adhering to the test tube, copper spiral and gas lead-in tube and add the washings to the insulating liquid in the flask.

The mixture is then allowed to stand in the dark for 24 h, at a temperature of $20 \text{ °C} \pm 5 \text{ °C}$, before filtering through a filter crucible (or membrane filter) previously dried to constant mass.

At the start of filtering only a small pressure drop should be used, to prevent the sludge passing through the filter. Cloudy filtrates should be passed through a second time.